L16 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2003 ACS on STN
AN 2000:384088 CAPLUS
DN 133:8186
TI Mircrocrack-free quartz glass jigs having large surface irregularities for semiconductor devices and their manufacture
IN Inaki, Kyoichi
PA Heraeus Quarzglas G.m.b.H. und Co. K.-G., Germany; Shin-Etsu Quartz Products Co., Ltd.
SO PCT Int. Appl., 15 pp.
CODEN: PIXXD2
DT Patent
LA English
IC ICM C03C017-22
ICS H01L021-68; C03C017-245; C03C017-02
CC 57-1 (Ceramics)
Section cross-reference(s): 76
FAN.CNT 1

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PI	WO 2000032529	A1	20000608	WO 1999-EP9261	19991129 <--
	W: US				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	JP 2000169165	A2	20000620	JP 1998-342157	19981201
	EP 1051363	A1	20001115	EP 1999-958136	19991129
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	US 6458445	B1	20021001	US 2000-601155	20000729
PRAI	JP 1998-342157	A	19981201		
	WO 1999-EP9261	W	19991129		

AB The title jig has large surface irregularities having a center line roughness Ra 2-30 .mu.m, a max. height Rmax 10-150 .mu.m, and a width of 10-500 .mu.m. The jig is manufd. by: forming an inorg. thin film on the surface of a quartz glass jig free from microcracks and having fine irregularities on the surface, and rinsing for plurality of times. Contamination is prevented when producing the semiconductor devices using the jig.
ST quartz glass jig surface irregularity semiconductor device
IT Films
(glass jig surface formed with; manuf. of microcrack-free quartz glass jigs having large surface irregularities for semiconductor devices for contamination prevention)
IT Jigs
Semiconductor devices
(manuf. of microcrack-free quartz glass jigs having large surface irregularities for semiconductor devices for contamination prevention)
IT 60676-86-0, Silica, vitreous
RL: DEV (Device component use); PRP (Properties); USES (Uses)
(manuf. of microcrack-free quartz glass jigs having large surface irregularities for semiconductor devices for contamination prevention)
IT 7440-21-3, Silicon, processes 7631-86-9, Silica, processes 12033-89-5, Silicon nitride, processes
RL: PEP (Physical, engineering or chemical process); TEM (Technical or engineered material use); PROC (Process); USES (Uses)
(thin film formed on glass jig; manuf. of microcrack-free quartz glass jigs having large surface irregularities for semiconductor devices for contamination prevention)
RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Anon; JP 10273339 A 1998 CAPLUS
(2) Heraeus Quarzglas; DE 19713014 A 1998 CAPLUS
(3) Shinetsu Handotai KK; EP 0704891 A 1996

(4) Shinetsu Quartz Prod Co Ltd; JP 08104541 A 1996 CAPLUS
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L16 ANSWER 2 OF 2 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN
AN 2000-431074 [37] WPIDS
DNN N2000-321737 DNC C2000-130921
TI Micro crack free quartz glass jig for use during chemical vapor deposition process for semiconductor device manufacture, has large irregularities possessing preset range of center line roughness, height and width.
DC L01 L03 U11
IN INAKI, K
PA (HERA) HERAEUS QUARZGLAS GMBH; (SHIN-N) SHINETSU QUARTZ PROD CO LTD; (SHIN-N) SHINETSU SEKIEI KK; (HERA) HERAEUS QUARZGLAS GMBH & CO KG
CYC 21
PI WO 2000032529 A1 20000608 (200037)* EN 15p C03C017-22 <--
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: US
JP 2000169165 A 20000620 (200040) 4p C03B020-00
EP 1051363 A1 20001115 (200059) EN C03C017-22
R: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE
US 6458445 B1 20021001 (200268) D06N007-04
ADT WO 2000032529 A1 WO 1999-EP9261 19991129; JP 2000169165 A JP 1998-342157 19981201; EP 1051363 A1 EP 1999-958136 19991129, WO 1999-EP9261 19991129; US 6458445 B1 WO 1999-EP9261 19991129, US 2000-601155 20000729
FDT EP 1051363 A1 Based on WO 200032529; US 6458445 B1 Based on WO 200032529
PRAI JP 1998-342157 19981201
IC ICM C03B020-00; C03C017-22; D06N007-04
ICS C03C015-00; C03C017-02; C03C017-245; H01L021-68
AB WO 200032529 A UPAB: 20000807
NOVELTY - Micro crack free quartz glass jig has many large irregularities on the surface. The regularities has a center line roughness in the range of 2-30 microns . The maximum height and width of the irregularities are in the range of 10-150 microns and 10-500 microns respectively.
DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for a method of producing quartz glass jig.
USE - Micro crack free quartz glass jig for use during chemical vapor deposition process for semiconductor device manufacture.
ADVANTAGE - The quartz glass jig having large irregularities on the surface are attained thus manufacture of semiconductor products free from contamination can be obtained.
DESCRIPTION OF DRAWING(S) - The figure shows the secondary electron image photograph of the surface of the quartz glass jig, when observed under a scanning electron microscope at a magnification of 50 times.
Dwg.1/4
FS CPI EPI
FA AB; GI
MC CPI: L01-L; L04-C12; L04-D09
EPI: U11-C09B; U11-F02A2

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(74) Agent: KÜHN, Hans–Christian; Heraeus Holding GmbH, Schutzrechte, Heraeusstrasse 12–14, D–63450 Hanau (DE).

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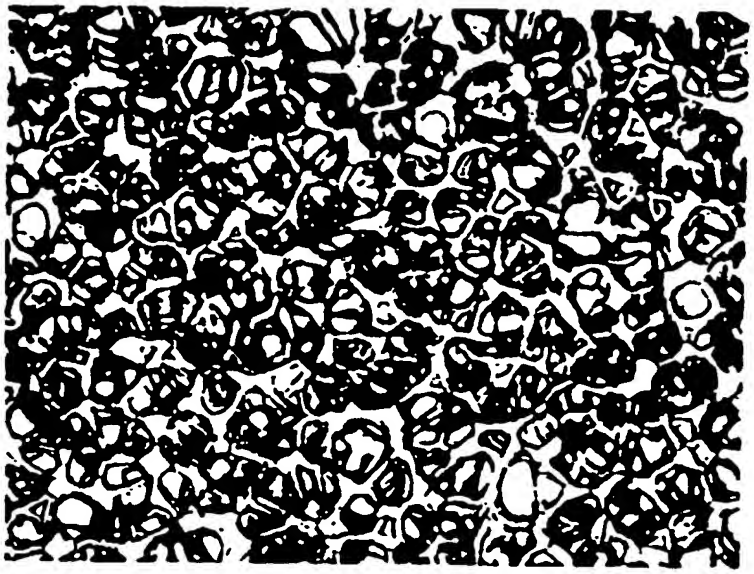
(54) Title: QUARTZ GLASS JIG HAVING LARGE IRREGULARITIES ON THE SURFACE AND METHOD FOR PRODUCING THE SAME

(57) Abstract

An object of the present invention is to provide a quartz glass jig free from microcracks in the oxide film formed by a CVD process and thelike even after rinsing for a plurality of times and which enables semiconductor products free from contamination due to the generation of particles. Another object of the present invention is to provide a production method thereof. The object above is accomplished by a quartz glass jig having large irregularities on the surface thereof, characterized in that said irregularities have a center line roughness Ra in the range of from 2 to 30 μm, a maximum heigt Rmax in the range of from 10 to 150 μm, and a width in the range of from 10 to 500 μm, and by a method which comprises forming an inorganic thin film on the surface of a quartz glass jig free from microcracks and having fine irregularities on the surface thereof, and thereafter rinsing it a plurality of times.



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Quartz Glass Jig Having Large Irregularities on the Surface and Method for Producing the Same

Detailed Description of the Invention

Industrial Field of Application

The present invention relates to a quartz glass jig and a method for producing the same; in further detail, it relates to a quartz glass jig free from microcracks and having large irregularities on the surface thereof, and to a method for producing the same.

Prior Art

Conventionally, high purity quartz glass jigs having a relatively high thermal resistance and high chemical resistance are widely used for the production of semiconductor devices. Furthermore, it is often the case that irregularities are intentionally provided on the surface of the jigs, and these irregularities are generally formed by frost treatment. However, a conventional frost treatment comprises shaving off the surface of the quartz glass jigs by spraying a powder of crystalline silicon dioxide. Thus, microcracks generate at the same time irregularities are formed, and this caused selective etching of the microcracks during the subsequent rinsing treatment using an aqueous solution of hydrogen fluoride. This led to problems such as the adhesion of etching solution or to the generation of particles which contaminate the semiconductor products.

As a means to solve th probl ms above, in JP-A-Hei10-273339 (the term „JP-A" as referred h rein signifies „an unexamined published Japanese patent application") is proposed a quartz glass jig having a rough surface formed by a structure comprising irregular elevations extended between the first upper level and the second lower level, the surface of which having an average height in the range of from 0.1 to 10 μm and protruded portions having an average width in the range of from 30 to 180 μm. In Japanese patent application JP-Hei-9-282757 is proposed a quartz glass jig having spherical or ellipsoidal irregularities on the surface thereof.

Problems the Invention is to Solve

When CVD process is carried out by using the former type of quartz glass jig above, however, although thin films free from the generation microcracks and in which the generation of particles is suppressed are obtained in the initial stage, the irregularities on the surface of the jig are lost with repeated rinsing, and there was found to cause a problem of forming microcracks on the thus vapor deposited thin film, which led to the generation of particles that contaminate the semiconductor products. Thus quartz glass jigs should be obtained capable of realizing thin films deposited by CVD process free from the formation of microcracks even after repeated rinsing and free from the generation of microcracks, thereby capable of obtaining semiconductor products free from contamination. The present invention has been accomplished based on these findings.

Thus, an object of the present invention is to provide a quartz glass jig having large irregularities on the surface thereof.

Furthermore, another object of the present invention is to provide a quartz glass jig which realizes an oxidation film formed by CVD process and the like free from the formation of microcracks even after repeated rinsing, and which is also free from causing contamination of s miconductor products attributed to the generation of particles.

Yet further object of the present invention is to provide a simple method for producing the quartz glass jig above.

Means for Solving the Problems

Accordingly, the present inv ntors hav_ intensively conducted studies to solve the problems above. As a result, the present invention which accomplishes the object above provides a quartz glass jig having large irregularities on the surface thereof, characterized in that said irregularities have a center line roughness Ra in the range of from 2 to 30μm, a maximum height Rmax in the range of from 10 to 150μm, and a width in the range of from 10 to 500μm on the surface of the quartz glass jig, and a method for producing the same.

The quartz glass jig according to the present invention is useful as a jig for use in the semiconductor industry, for example, a furnace core tube, a boat for mounting wafers thereon, etc., which is a quartz glass jig free from microcracks, and which comprises a surface on which irregularities are partly or entirely formed with a center line roughness Ra in the range of from 2 to 30 μm, preferably, from 10 to 30 μm, a maximum height Rmax in the range of from 10 to 150 μm, preferably, from 50 to 150 μm, and a width in the range of from 10 to 500μm. Because the quartz glass jig comprises such large irregularities and is free from microcracks, there is no change in surface roughness even after an etching treatment using a 5 % hydrofluoric acid, and maintains the irregularities without change in center line roughness and in the maximum height. Accordingly, even if these quartz glass jigs are used in CVD processes and are subjected to repeated rinsing, the oxide films formed thereon remain free from microcracks and do not contaminate semiconductor products attributed to the generation of particles. Particularly preferred is, as shown in Fig. 1, that the irregularities are formed from a plurality of slopes, and the apex at which the slopes cross with each other forms an edge line, for example, the irregularities are formed in a shape similar to the roofs such as a gable roof or a hip roof. The slopes above may be planar or curved planes. The term „rinsing" as referred herein signifies rinsing using an aqueous hydrofluoric acid solution, or a mixed solution of hydrofluoric acid with an inorganic acid such as nitric acid or sulfuric acid. If the irregularities have a center line roughness Ra of lower than 2 μm, the thin film is subject to the generation of microcracks, and if the irregularities yield a center line roughness Ra exceeding 30 μm, it becomes difficult to form large irregularities. Similar to the case of center line roughness Ra, irregularities having a maximum Rmax or a width falling out of the aforementioned ranges cannot form a favorable quartz glass jig.

Futhermore, it has been found that the quartz glass jig having large irregularities on the surface thereof can asisy be produced by repeating the st ps of forming an inorganic thin film on the surface of a quartz glass jig free from microcracks but having fine irregularities, followed by rinsing.

The quartz glass jig having large irregularities on the surface thereof can be produced by first forming a thin film of silicone and the like in accordance with, for instance, the d scription given in the specification of Japanese patent application JP-Hei-9-282757, and then subjecting the resulting product to an etching treatment using an aqueous solution of hydrofluoric acid, or by immersing the quartz glass jig into a solution consisting of a mixture of hydrofluoric acid, ammonium fluoride, and acetic acid in accordance with the frost treatment as described in JP-A-Hei10-273339, thereby forming fine irregularities free from microcracks on the surface by using the precipitates of ammonium silicofluoride, and then repeating at least twice a step of coating the surface of the jig with an inorganic thin film followed by rinsing until there is formed irregularities having a center line roughness Ra in a range of 2 to 30μm, preferably, in a range of 10 to 30 μm, a maximum height Rmax in a range of 10 to 150μm, preferably, in a range of 50 to 150 μm, and a width in a range of 10 to 500μm. Unlike the case of a conventional sandblasting method, in which microcracks are generated and in which aqueous hydrofluoric acid intrudes into the microcracks to form acute front ends due to the release thereof into oval shapes, the above method of forming irregularities comprising coating an inorganic thin film and rinsing, the apices of the irregularities are formed in the shape of smooth slopes. As the inorganic thin films above, there can be mentioned thin films of at least one type of material selected from silicon compounds, silicon nitride compounds, silicon oxide compounds, and silica glasses. The reason why large irregularities are formed by thus rinsing after forming an inorganic thin film on the surface of a quartz glass having fine irregularities and free from microcracks is that, presumably, visually non-discernible fine microcracks are formed at the time the thin film is formed on the indented portions of the fine irregularities, and the rinsing solution intrudes along the fine microcracks as to selectively etch and enlarge the irregularities. The inorganic thin film is preferably formed in a range of from 0.1 to 100μm. If the thin film is formed at a thickness less than 0.1 μm, there is no increase in roughness nor formation of large irregularities on rinsing. If the thin film is formed at a thickness exceeding 100μm, visually observable linear microcracks form, and these microcracks reach to the quartz glass jig and develop in such a manner to surround the quartz glass jig Thus, it is not preferred because quartz glass tend to fall off.

Embodiment of the Present Invention

An embodiment of the present invention is described below by way of xamples and drawings, but it should be understood that the present invention is not limited thereto.

Brief Explanation of the Drawings

Fig. 1: Fig. 1 is a secondary lectron image photograph of the surface of a quartz glass jig according to the present invention observed under a scanning electron microscope at a magnification of 50 times.

Fig. 2: Fig. 2 is a secondary electron image photograph of the surface of a quartz glass jig having formed thereon a poly-Si film and rinsed four times, observed under a scanning electron microscope at a magnification of 50 times.

Fig. 3: Fig. 3 is a profile obtained by measuring the surface of a quartz glass jig according to the present invention using a surface roughness meter.

Fig. 4: Fig. 4 is a profile obtained by using a surface roughness meter on the surface of a quartz glass jig having formed thereon a poly-Si film and rinsed four times.

Example 1

A quartz glass tube was immersed into a solution obtained by mixing about 24 % by weight of an aqueous 50 % hydrofluoric acid solution, about 17 % by weight of ammonium fluoride, about 35 % by weight of an aqueous 100 % acetic acid solution, and about 24 % by weight of water, to thereby obtain fine crystals of ammonium silicon fluoride as precipitates. The quartz glass tube thus obtained yielded a center line roughness Ra of 0.5μm, a maximum height of the irregularities Rmax of 2μm, but no microcracks were observed visually. A poly Si film was vapor deposited on the surface of the thus obtained quartz glass tube at a thickness of 10 μm, and the rinsing process using a mixed solution of hydrofluoric acid and nitric acid was repeated four times. Referring to Fig. 1, on the surface of the resulting quartz glass tube were formed irregularities consisting of a plurality of slopes, and an edge line was formed at the apex at which the slopes cross each other. Figure 1 above is a secondary electron image obtained by scanning electron microscope at a magnification of 50 times. The surface of the quartz glass jig thus obtained was measured using a surface roughness meter (Model Surfcom 300B, manufactured by Tokyo Seimitsu Co., Ltd.), and the results are shown in Fig. 3, in which the longitudinal direction (x-axis) is magnified by 500 times, and the horizontal direction (y-axis) is magnified by 300 times, the latter characterizing the roughness R. Referring to Fig. 3, the surface of the quartz glass jig yields a center line roughness Ra of 13μm, a maximum height of the irregularities Rmax of 80 μm, and an av rage width of the irregularities of 100μm. The thus ob-

tained quartz glass tube was used in a CVD process for silicon wafers, and found no gen ration of particles even after rinsing for four times.

Comparative Example 1

A quartz glass tube was immersed into a solution obtained by mixing about 24 % by weight of an aqueous 50 % hydrofluoric acid solution, about 17 % by weight of ammonium fluoride, about 35 % by weight of an aqueous 100 % acetic acid solution, and about 24 % by weight of water, to thereby obtain fine crystals of ammonium silicon fluoride as precipitates. The quartz glass tube thus obtained yielded a center line roughness Ra of 0.5μm, a maximum height of the irregularities Rmax of 2 μm. The resulting quartz glass tube was directly used in a CVD process for silicon wafers, but particles generated after rinsing for four times, and it was confirmed that contamination occurred on the silicon wafer.

Comparative Example 2

A poly Si film was vapor deposited on the surface of a transparent quartz glass tube at a thickness of 10 μm, and the resulting product was subjected to four times of rinsing using a mixed solution of hydrofluoric acid and nitric acid. As shown in Fig. 2, numerous linear microcracks capturing the glass were found to generate on the surface of the thus obtained quartz glass tube. Figure 2 is a secondary electron micrograph obtained by a scanning electron microscope operated at a magnification of 50 times. Furthermore, the surface of the resulting quartz glass tube was measured using a surface roughness meter in a manner similar to that described in Example 1, and a graph enlarged by 500 times in the vertical direction and by 300 times in the horizontal direction is shown in Fig. 4. Referring to Fig. 4, the center line roughness Ra was 6 μm, and the maximum height of the irregularities Rmax was 40μm. The quartz glass tube thus obtained was used in a CVD process for silicon wafers, but the test was interrupted due to the generation of abnormal particles.

Effect of the Invention

The quartz glass jig according to the present invention has large irregularities on the surface thereof and yet, free from microcracks. When the quartz glass jig is used in the CVD processes for producing semiconductor products, it can be reus d in CVD processes v n after it is rinsed for a plurality of times without forming any microcracks and th like on oxide films, and without contaminating the semiconductor products attributed to th formation of particles.

Fine irregularities free from microcracks can be formed on the surface of the quartz glass of the quartz glass jigs, and it can be asily produced by repeating, for a plurality of times, the process of forming a coating of an inorganic thin film on the surface thereof and rinsing it thereafter. Thus, the present invention is of high value in the industry.

Claims

1. A quartz glass jig having large irregularities on the surface thereof, characterized in that said irregularities have a center line roughness Ra in the range of from 2 to 30μm, a maximum height Rmax in the range of from 10 to 150μm, and a width in the range of from 10 to 500 μm.

2. A quartz glass jig as claimed in Claim 1, wherein said irregularities have a center line roughness Ra in the range of from 10 to 30 μm, a maximum height Rmax in the range of from 50 to 150 μm, and a width in the range of from 10 to 500μm.

3. A quartz glass jig as claimed in Claim 1 or 2, wherein said irregularities are formed from a plurality of slopes, and the apex at which the slopes cross with each other forms an edge line.

4. A quartz glass jig as claimed in Claims 1 to 3, wherein the quartz glass jig having large irregularities on the surface thereof is a jig for use under an atmosphere for forming a thin film by reacting gaseous starting materials.

5. A method for producing a quartz glass jig having large irregularities on the surface thereof, which comprises repeating several times a process of forming an inorganic thin film on the surface of a quartz glass jig free from microcracks and having fine irregularities on the surface thereof, and thereafter rinsing it.

6. A method for producing a quartz glass jig having large irregularities on the surface thereof as claimed in Claim 5 wherein the inorganic thin film is formed from at least one type selected from the group consisting of a silicon compound, a silicon nitride compound, a silicon oxide compound, and a silica based glass.

7. A method for producing a quartz glass jig having large irregularities on the surface thereof as claimed in Claim 5 or in Claim 6, wherein the thickness of th thin film is in the range of from 0.1 to 100 μm.

Fig. 1

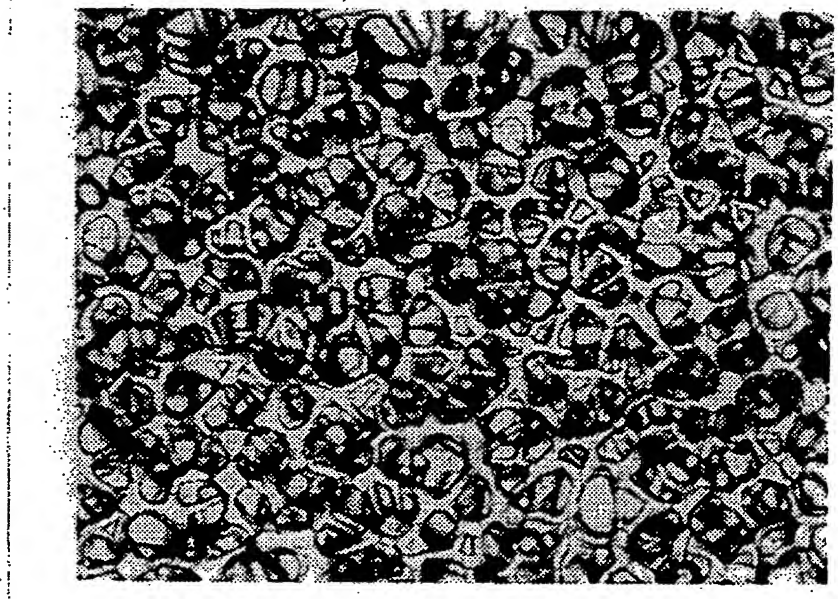


Fig. 3

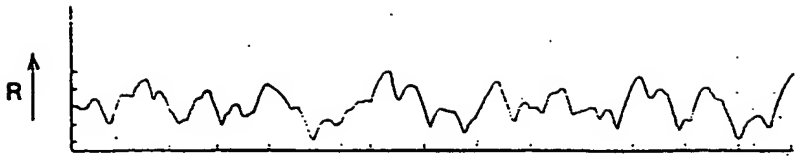


Fig. 2

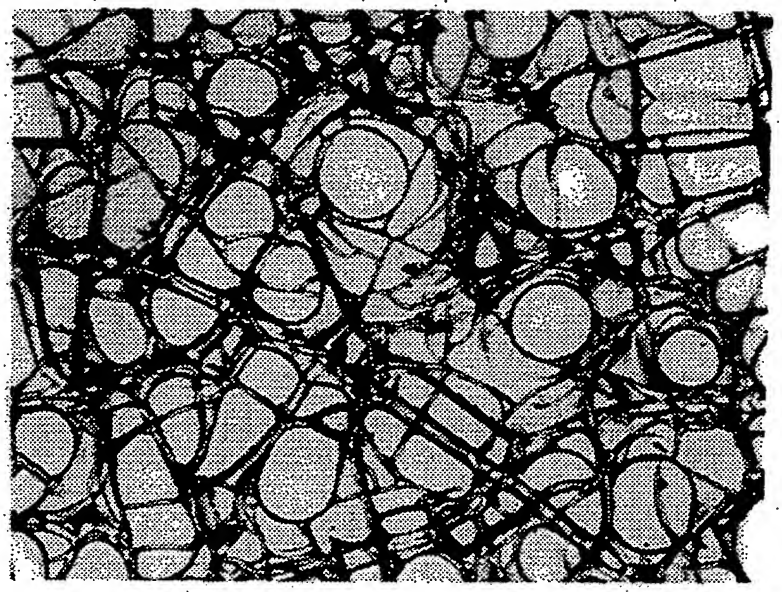
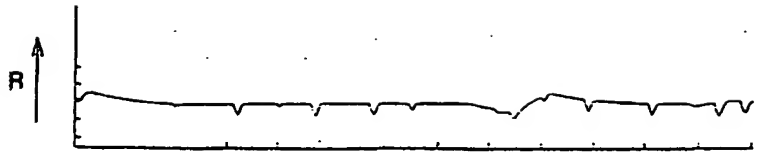


Fig. 4



INTERNATIONAL SEARCH REPORT

Int ional Application No
PCT/EP 99/09261

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C03C17/22 H01L21/68 C03C17/245 C03C17/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C03C H01L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 704 891 A (SHINETSU HANDOTAI KK) 3 April 1996 (1996-04-03) page 4, line 12 -page 5, line 47; claims	1
A		2-7
X	PATENT ABSTRACTS OF JAPAN vol. 1996, no. 08, 30 August 1996 (1996-08-30) & JP 08 104541 A (SHINETSU QUARTZ PROD CO LTD), 23 April 1996 (1996-04-23) abstract	1
A		2-7

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[X] Further documents are listed in the continuation of box C. [X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
28 February 2000	06/03/2000

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

Inte ional Application No
PCT/EP 99/09261

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 015, no. 440 (C-0883), 11 November 1991 (1991-11-11) & JP 03 187954 A (TOSHIBA CERAMICS CO LTD), 15 August 1991 (1991-08-15) abstract	1
A		2-7
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